LiGA Fabricated High Aspect Ratio Nickel Gas Chromatograph Columns as a Step Towards a Portable and Fast GC Instrument

Abhinav Bhushan, Dawit Yemane, Jost Goettert, Center for Advanced Microstructures and Devices (CAMD) , Louisiana State University, Baton Rouge LA

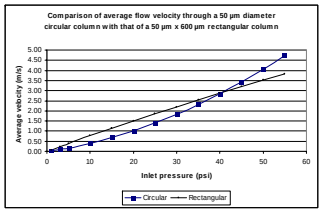
Edward Overton, Department of Environmental Studies, Louisiana State University, Baton Rouge, LA

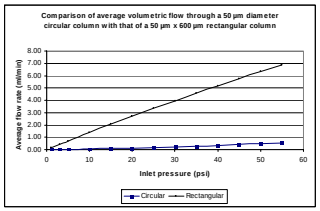
Introduction

A GC-MS analysis is considered the "gold standard" in scientific analysis. Combining the micro-GC system with a miniature mass spectrometer will lead to a truly portable and fast instrument capable of confirmed testing of compounds. This project involves using the LiGA micromachining technique to build high aspect ratio gas chromatograph columns to be used as part of a miniaturized GC system. The columns are 30-50 µm wide, 400-650 µm tall (aspect-ratios of 10-15), 0.5-2 m long and cover a spatial area of 1-2 cm². LiGA enables efficient integration of such functions as miniature detectors, heaters for temperature programming, and multi-stage pre-concentration directly onto the column chip to form a compact handheld system with enhanced performance with of reduced power consumption and increased speed and resolution of analysis.

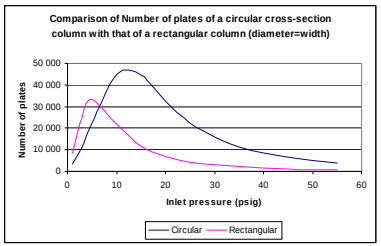
Advantages of High Aspect Ratio Rectangular Columns

Theory suggests that the resolution of the separation is governed by the column width, while the volumetric flow rate and hence the sample carrying capacity depends upon the cross-sectional area of the column. For instance, a 50 µm wide by 600 µm tall rectangular cross-section column will have a flow rate equivalent to a 90 µm diameter circular capillary column, while having the resolution of a 50 µm column. In the figure below, the flow between a circular capillary column is compared to that of a high aspect ratio (HAR) column. While the average flow velocities are of the same order, the volumetric flow rate for the HAR column is much more than that for the capillary column, showing the higher sample carrying capacity of the HAR column.



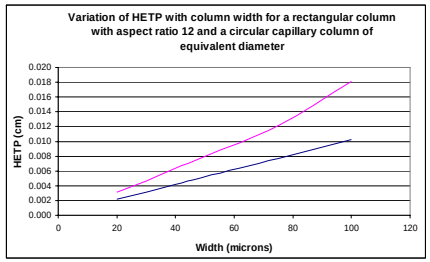


Sample capacity$_{rectangular}$>> Sample capacity$_{circular}$



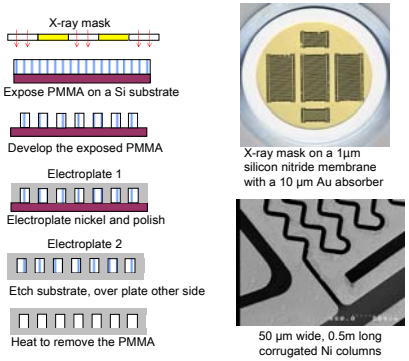
N of plates$_{rectangular}$~ N of plates$_{circular}$
(width w) (diameter w)

HETP$_{rectangular}$ < HETP$_{circular}$



LIGA Fabrication Process

The fabrication process includes x-ray lithography and two-sided nickel electroplating. A gold absorber x-ray mask is used to define the pattern on a photopolymer using X-rays.



X-ray mask on a 1µm silicon nitride membrane with a 10 µm Au absorber


Overview of a 2m long column with top/bottom inlets

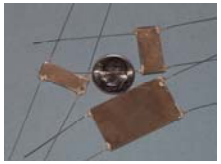
50 µm wide, 0.5m long corrugated Ni columns

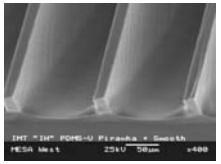

Sealed nickel columns showing the extremely parallel side walls

Column Connection and Coating

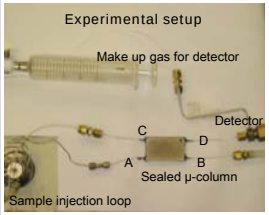
After completion of the fabrication process metal tubes are soldered into the appropriate slots to build a sealed column system. These tubes are also used to load the stationary phase into the column. Coating the microfabricated column is complex. Some of the challenges are:

- Deactivation and cleaning of the metal column surface;
- Pooling of the stationary phase in the corners;
- Uniform deposition of stationary phase on the large surface area.

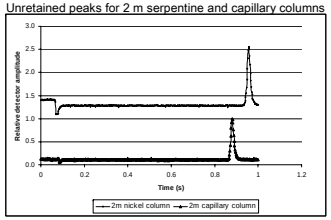

Metal tubing attached to the nickel columns using a silver paste which can withstand 300 C


Stationary phase pooling in the corners of the column. (courtsey Sandia Natl. Labs)

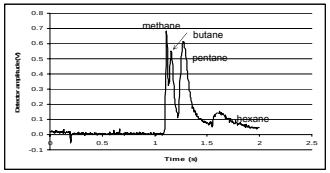
Column Testing



Unretained peaks for 2 m serpentine and capillary columns



The separation of four compounds using the LiGA nickel column coated with RTX-1 is shown. The experiment was performed at 25 °C at 30 psi head pressure of the carrier gas. The broad peaks suggest that either the stationary phase has pooled in the corners

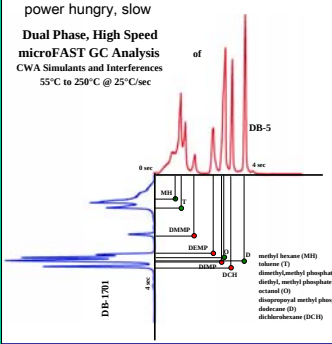


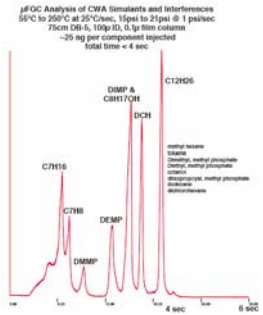
Column Testing using the microFast GC

The microFast GC developed by Analytical Specialists Inc., Baton Rouge, Louisiana is capable of doing complex analysis in a few seconds. Because of the modularity and ease of operation of the device, it being used as the experimental bench for testing the microfabricated columns.


HP5890: Bench top instrument, power hungry, slow

microFast GC: portable, extremely fast, runs on a 12V/24V battery



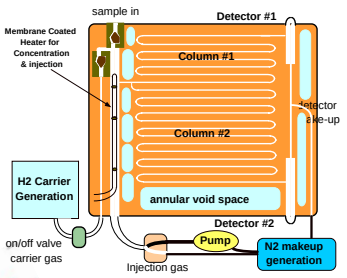


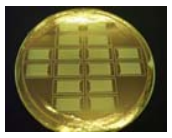
Future Outlook

Additional functionality can be incorporated in the column chip:

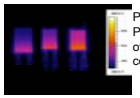
- Lower mass will reduce the power required to heat the chip for temperature programming. Thus low power thin film heaters can be integrated on the column
- On-chip check valves will reduce dead volume
- A SPME type insert as a PC can be integrated right into the column space

A lot of these modules are currently being incorporated into the column chip.




Thin film heaters on a polyimide substrate to integrate with the column


ANSYS simulation of gradient temperature distribution on a column chip


Profiled Temperature Programming (PTP) of a 2m micro-column chip


The future?

Acknowledgements

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